

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Syuhei OKUDE et al.

Application No.: 10/584,412

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For: POLARIZING PLATE PROTECTIVE FILM,
REFLECTION PREVENTIVE POLARIZING
PLATE AND OPTICAL PRODUCT

Examiner: E. A. Robinson

DECLARATION UNDER 37 CFR § 1.132

Commissioner for Patents

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Sir:

I, Kiroshi Yakabe, declare the following.

I am fully knowledgeable of the disclosure of the above-identified application and the field of art of the present invention. I have read and understand the Office Action dated February 11, 2008 and the references cited therein to Shoshi et al. '188 (U.S. 2003/0104188), Murakami et al. '900 (U.S. 5,681,900), and Nakamura et al. '929 (U.S. 2001/0035929).

It is my opinion that the presently claimed polarizing plate protective film which comprises a low-refractive-index layer including a hollow microparticle having a shell and a cavity formed inside the shell, wherein the cavity is completely enclosed by the

shell, has unexpectedly superior properties to polarizing plate protective films which comprise a low-refractive-index layer including porous microparticles such as those disclosed by Shoshi et al. '188. It is also my opinion that the presently claimed polarizing plate protective film which comprises a low-refractive-index layer including a hollow microparticle having a shell and a cavity formed inside the shell, wherein the cavity is completely enclosed by the shell, is not made obvious by Shoshi et al. '188 Murakami et al. '900 and Nakamura et al. '929.

The following experiments were performed by me or under my direct supervision.¹

(Preparation of base film 1A)

100 parts by weight of a norbornene polymer (hydrogenated product of ring-opening copolymer of norbornene monomers, "ZEONOR 1430" manufactured by Zeon Corporation, glass transition temperature (T_g) = 145°C) was mixed with 0.2 parts by weight of a phenol aging preventive (pentaerythrityl-tetrakis [3-(3,5-di-tertiarybutyl-4-hydroxyphenyl)propionate]). The mixture was kneaded using a biaxial mixer, and the resulting strand (rod-like molten resin) was cut using a strand cutter to obtain a pellet-shaped (particulate) molding material.

The resulting pellets were dried at 110°C for four hours using a hot air dryer in which air was circulated. Using a T-die film melt extrusion molding machine having a resin melt kneader equipped with a screw with a diameter of 65 mm that provided with a leaf disk polymer filter (filtration accuracy: 30 μ m), the dried pellets were extruded using a T-shaped die with a width of 350 mm, in which chrome plating with a surface roughness Ra of 0.15 μ m was provided on the inner surface, at a molten resin

¹ Please note that the numbering/lettering used herein is not perfectly sequential, since I have retained the numbering/lettering of the experiments which coincide with the Examples in the present specification.

temperature of 260°C and a die temperature of 260°C. The extruded sheet-shaped norbornene polymer was caused to adhere to a first cooling drum (diameter: 250 mm, temperature: 135°C, circumferential speed R_1 : 10.05 m/min), and then transferred to a second cooling drum (diameter: 250 mm, temperature: 125°C, circumferential speed R_2 : 10.05 m/min) and a third cooling drum (diameter: 250 mm, temperature: 100°C, circumferential speed R_3 : 9.98 m/min) while causing the polymer to adhere to the drum to obtain a base film 1A with a length of 300 m and a thickness of 40 μm). The resulting long base film 1A was wound in the shape of a roll. The base film 1A had a volatile content of 0.01 wt% or less and a saturated water absorption of 0.01 wt% or less.

(Preparation of hard coating material 1)

30 parts of a hexafunctional urethane acrylate oligomer ("NK Oligo U-6HA" manufactured by Shin-Nakamura Chemical Co., Ltd.), 40 parts of butyl acrylate, 30 parts of isobornyl methacrylate ("NK Ester IB" manufactured by Shin-Nakamura Chemical Co., Ltd.), and 10 parts of 2,2-dimethoxy-1,2-diphenylethan-1-one were mixed using a homogenizer to prepare a hard coating material 1 which is UV-curable resin composition.

(Preparation of hard coating material 2)

100 parts by weight of an antimony pentoxide modified alcohol sol (manufactured by Catalysts & Chemicals Industries Co., Ltd., solid content: 30 wt%), 10 parts by weight of UV-curable urethane acrylate ("UV7000B" manufactured by Nippon Synthetic Chemical Industry Co., Ltd.), and 0.4 parts by weight of a photoinitiator ("Irgacure 184" manufactured by Ciba Specialty Chemicals Inc.) were mixed to prepare a UV-curable hard coating material 2.

(Production Example 1) Production of hard coating layer stacked film 2A

The upper and lower sides of the *base film 1A* were subjected to a corona discharge treatment for three seconds using a high frequency transmitter (corona generator “HV05-2” manufactured by Tantec) at an output voltage of 100%, a power of 250 W, an electrode length of 240 mm (wire electrode with a diameter of 1.2 mm), and a work electrode interval of 1.5 mm to modify the *base film 1A* so that the surface tension was 0.072 N/m. The *hard coating material 1* was continuously applied to the *base film 1A* using a die coater so that the thickness of the hard coating layer after curing was 5 μm . After drying the applied *hard coating material 1* at 80°C for five minutes, the hard coating material was cured by applying ultraviolet rays (dose: 300 mJ/cm^2) to obtain a *hard coating layer stacked film 2A*.

(Production Example 2) Production of hard coating layer stacked film 2B

A *hard coating layer stacked film 2B* was obtained in the same manner as in Production Example 1 except for using a triacetyl cellulose (TAC) film with a thickness of 40 μm (*base film 1B*, “KC4UX2M” manufactured by Konica Minolta, $T_g=120^\circ\text{C}$, saturated water absorption=4.5 wt%, volatile content=6.0 wt%).

(Production Example 3) Production of hard coating layer stacked film 2C

A *hard coating layer stacked film 2C* was obtained in the same manner as in Production Example 1 except for using a polyethylene terephthalate film with a thickness of 40 μm (*base film 1C*, “Lumirror T60” manufactured by Toray Industries Inc.).

(Production Example 4) Preparation of coating composition 1

A tetramethoxysilane oligomer (“Methyl Silicate 51” manufactured by Colcoat Co., Ltd.), methanol, water, and a 0.01N hydrochloric acid solution were mixed at a

weight ratio of 21:36:2:2. The mixture was stirred at 25°C for two hours in a high-humidity bath to obtain a silicone resin with a weight average molecular weight of 850. A hollow silica isopropanol dispersion sol (manufactured by Catalysts & Chemicals Industries Co., Ltd., solid content: 20 wt%, average primary particle diameter: about 35 nm, shell thickness: about 8 nm) was added to the silicone resin as hollow silica microparticles so that the weight ratio of “hollow silica microparticle/silicone resin (converted into condensation compound)” was 7:3 (solid content). The mixture was diluted with methanol so that the total solid content was 1 wt% to obtain a coating composition 1.

(Production Example 5) Preparation of coating composition 2

A coating composition 2 was prepared in the same manner as in the preparation of the coating composition 1 except that the hollow silica isopropanol dispersion sol was added so that the weight ratio of “hollow silica microparticle/silicone resin (converted into condensation compound)” was 8:2 (solid content) and silica methanol (“PMA-ST” manufactured by Nissan Chemical Industries, Ltd., and average particle diameter: 10 to 20 nm) was added as silica microparticles in which a cavity is not formed inside the shell in an amount of 5% (SiO₂ conversion solid content) of the total solid content of the coating composition.

(Production Example A) Preparation of coating composition 3

A tetramethoxysilane oligomer ("Methyl Silicate 51" manufactured by Colcoat Co., Ltd.), methanol, water, and a 0.01N hydrochloric acid solution were mixed at a weight ratio of 21:36:2:2. The mixture was stirred at 25°C for two hours in a high-humidity bath to obtain a silicone resin with a weight average molecular weight of 850. And porous silica sol containing porous silica microparticles (manufactured by Catalysts & Chemicals Industries Co., Ltd., solid content: 10 wt%, average primary particle diameter: about 50 nm, refractive index:1.34) was added to the silicone resin so that the weight ratio of "porous silica microparticle/silicone resin (converted into condensation compound)" was 7:3 (solid content). The mixture was diluted with methanol so that the total solid content was 1 wt% to obtain a coating composition 3.

(Example 1)

After allowing the coating composition 1 obtained in Production Example 4 to stand for one hour after the preparation, the composition 1 was applied to the hard coating layer stacked film 2A using a wire bar coater to form a coating with a thickness of about 100 nm. After allowing the coating to dry for one hour, the coating was heated at 120°C for 10 minutes in air to obtain a polarizing plate protective film 3A on which the cured coating was formed.

(Example 2)

A polarizing plate protective film 3B on which a cured coating was formed was obtained in the same manner as in Example 1 except for using the coating composition 2 obtained in Production Example 5.

(Example 3)

A polarizing plate protective film 3C on which a cured coating was formed was obtained in the same manner as in Example 2 except for using the hard coat stacked film 2B obtained in Production Example 2.

(Example 4)

A polarizing plate protective film 3D on which a cured coating was formed was obtained in the same manner as in Example 2 except for using the hard coat stacked film 2C obtained in Production Example 3.

(Example 5)

A polarizing plate protective film 3E on which a cured coating was formed was obtained in the same manner as in Example 1 except for using the hard coating material 2 instead of the hard coating material 1.

(Comparative Example 3)

After allowing the coating composition 3 to stand for one hour after the preparation, the composition 3 was applied to the hard coating layer stacked film 2A using a wire bar coater to form a coating with a thickness of about 100 nm. After allowing the coating to dry for one hour, the coating was heated at 120°C for 10 minutes in air to obtain a polarizing plate protective film 3H on which the cured coating was formed.

The refractive indices of the low-refractive-index layer and the hard coating layer, the reflectance, the outward appearance of the film after the steel wool test, the total light transmittance and the haze before and after the steel wool test, and the warping rate (%) were measured using the polarizing plate protective films 3A to 3E of Examples 1 to 5 and the polarizing plate protective film 3H of Comparative Example 3.

The measurement results for the polarizing plate protective films 3A to 3E and 3H are summarized in Table 1 below.

[Table 1]

	Polarizing plate protective film	Refractive index of hard coating layer	Refractive index of low-refractive-index layer	Reflectance	Outward appearance of film after steel wool test	Before steel wool test		After steel wool test		Warping rate (%)
						Total light transmittance (%)	Haze (%)	Total light transmittance (%)	Haze (%)	
Example 1	3A	1.53	1.28	0.2	Excellent	95.5	0.47	95.0	0.52	0.6
Example 2	3B	1.53	1.25	0.2	Excellent	96.2	0.51	95.3	0.55	0.5
Example 3	3C	1.53	1.25	0.3	Good	94.9	0.55	94.4	0.58	0.9
Example 4	3D	1.53	1.25	0.2	Good	95.7	0.49	94.9	0.53	1.0
Example 5	3E	1.62	1.28	0.2	Excellent	95.5	0.50	94.8	0.53	0.6
Comparative Example 3	3H	1.62	1.355	0.5	Bad	94.7	0.5	93.6	8.26	0.6

The polarizing plate protective films 3A to 3E were prepared with a low-refractive-index layer including a hollow microparticle having a shell and a cavity formed inside the shell, wherein the cavity is completely enclosed by the shell, as presently claimed.

As shown in Table 1, the polarizing plate protective films 3A to 3E of Examples 1 to 5 showed a low refractive index of the low-refractive-index layer and exhibited a low reflectance. This suggests that the polarizing plate protective films 3A to 3E are useful as an optical film having an antireflection function.

In the polarizing plate protective films 3A to 3E of Examples 1 to 5, no scratches were observed on the surface with the naked eye after the steel wool test. Moreover, the changes in total light transmittance and haze before and after the steel wool test were small. Specifically, the polarizing plate protective films 3A to 3E exhibited excellent scratch resistance.

The polarizing plate protective films 3A to 3E of Examples 1 to 5 also exhibited a small warping rate.

This is distinct from the polarizing plate protective film 3H which was prepared with a low-refractive-index layer including porous microparticle, such as those taught by Shoshi et al. '188. As shown in Table 1, the haze value of the polarizing plate protective film 3H using the porous silica microparticles was remarkably increased after the steel wool test as compared with that of the polarizing plate protective films 3A-3E using the hollow silica isopropanol dispersion sol. It was found that the scratch resistance of the polarizing plate protective film 3H was inferior to that of the polarizing plate protective film 3A-3E.

In conclusion, a polarizing plate protective film having an excellent antireflection effect and scratch resistance could be obtained by using the hollow silica microparticles, as presently claimed.

It is my opinion that the presently claimed polarizing plate protective film which comprises a low-refractive-index layer including a hollow microparticle having a shell and a cavity formed inside the shell, wherein the cavity is completely enclosed by the shell, has unexpectedly superior properties to polarizing plate protective films which comprise a low-refractive-index layer including a porous microparticles such as those disclosed by Shoshi et al. '188. It is also my opinion that the presently claimed polarizing plate protective film which

comprises a low-refractive-index layer including a hollow microparticle having a shell and a cavity formed inside the shell, wherein the cavity is completely enclosed by the shell, is not made obvious by Shoshi et al. '188 Murakami et al. '900 and Nakamura et al. '929.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Hiroshi Yakabe

Signature

July 18, 2008

Date

Hiroshi YAKABE

Name in Print